INVESTIGATION OF ALTERNATIVE APPROACHES FOR CLEANING MOTT POROUS METALS FILTERS

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Summary

The Department of Energy selected Caustic Side Solvent Extraction (CSSX) as the preferred cesium removal technology for Savannah River Site (SRS) waste. As a pretreatment step for the CSSX flowsheet, the incoming salt solution that contains entrained sludge is contacted with monosodium titanate (MST) to adsorb strontium and select actinides. The resulting slurry is filtered to remove the sludge and MST. Filter fouling occurs during this process. At times, personnel can increase the filtrate rate by backpulsing or scouring. At other times, the filtrate rate drops significantly and only chemical cleaning will restore filter performance. The current baseline technology for filter cleaning uses 0.5 M oxalic acid. The Salt Processing Project (SPP) at SRS, through the Tanks Focus Area, requested an evaluation of other cleaning agents to determine their effectiveness at removing trapped sludge and MST solids compared with the baseline oxalic acid method.

A review of the technical literature identified compounds that appear effective at dissolving solid compounds. Consultation with the SPP management team, engineering personnel, and researchers led to a selection of oxalic acid, nitric acid, citric acid, and ascorbic acid for testing. Tests used simulated waste and actual waste as follows. Personnel placed simulated or actual SRS High Level Waste sludge and MST in a beaker. They added the selected cleaning agents, stirred the beakers, and collected supernate samples periodically analyzing for dissolved metals.

The conclusions from this work follow.

- 0.5 M oxalic acid and 4.0 M nitric acid proved most effective at dissolving iron, aluminum, and titanium. The 0.5 M oxalic acid solution dissolved 23 44 % of the iron, 11 41 % of the aluminum, and 98 100 % of the titanium calculated to be present in the solids used in the tests. The 4 M nitric acid solution dissolved 15 32 % of the iron, 13 47 % of the aluminum, and 67 100% of the titanium calculated to be present in the solids used in the tests. The citric acid solution dissolved 1 % of the iron, 7 % of the aluminum, and 17 % of the titanium. The ascorbic acid solution dissolved 8 % of the iron, 5 % of the aluminum, and 2 % of the titanium.
- 0.5 M oxalic acid proved more effective at dissolving iron, titanium, and silicon.
- 4 M nitric acid proved more effective at dissolving aluminum.
- Decreasing the concentration of oxalic acid or nitric acid decreased the amount of solids dissolved. Decreasing the oxalic acid concentration to 0.25 M reduced the amount of iron (from 23 to 10 %), aluminum (from 15 to 10 %), and titanium (from 100 to 56 %) dissolved. Decreasing the nitric acid concentration from 4 M to 0.5 M decreased the amount of iron (from 18 to 3 %), aluminum (from 19 to 9 %), and titanium (from 89 to 7 %) dissolved.

Based on these studies, the SPP should maintain 0.5 M oxalic acid as the baseline cleaning technology for the Salt Waste Processing Facility, and the Alpha Removal Project should use 0.5 M oxalic acid as their baseline cleaning solution.

Introduction

The Department of Energy selected CSSX as the preferred cesium removal technology for Savannah River Site waste. As a pretreatment step for the CSSX flowsheet, the incoming salt

solution that contains entrained sludge is contacted with MST to adsorb strontium and select actinides. The resulting slurry is filtered to remove the sludge and MST. The filtrate is processed through the solvent extraction system to remove cesium. Filter fouling occurs during this process. At times, personnel can increase the filtrate rate by backpulsing or scouring. At other times, the filtrate rate drops significantly and only chemical cleaning will restore filter performance. The current baseline technology for filter cleaning uses 0.5 M oxalic acid. In contrast, the baseline technology to clean crossflow filters for the Hanford Waste Treatment Plant uses 2 M nitric acid. ²

The Salt Processing Project (SPP) at SRS, through the Tanks Focus Area, requested the authors to perform an evaluation of other cleaning agents to determine their effectiveness at removing trapped sludge and MST solids compared with the baseline oxalic acid method.

The cleaning studies used simulated waste and actual waste. Simulant tests served as a screening tool, because of the cost of actual waste tests and the limited availability of actual waste samples.

Approach

The authors reviewed the technical literature to identify compounds that are effective at dissolving compounds such as iron, aluminum, silicon, and titanium.³⁻²⁰ The review identified the following cleaning agents as candidates.

- sodium hydroxide, sodium nitrate, and triethanolamine³
- citric acid^{4,5,8,9,11,16,17}
- LiOH, KOH, fluoride, and phosphate⁶
- hydrochloric acid^{7,14,15}
- nitriloacetic acid^{8,9}
- ascorbic acid^{9,11}
- EDTA^{8,9,12,16,17}
- pyridine dicarboxylic acid⁹
- catechol^{8,13}

- tributyl phosphate (TBP)¹⁴
- pyrophosphate¹⁶
- sodium hydroxide and Pluronic L62 at 100 °C
- mercaptocarboxylic acid¹⁷
- cysteine^{17,18}
- phenolics^{17,19}
- thioglycolic acid^{17,20}

We eliminated hydrochloric acid from consideration because of its corrosivity. We eliminated triethanolamine because of concerns about adding ammonia compounds to the high level waste system. The review panel considered pyrophosphate, TBP, LiOH, KOH, fluoride, and phosphate unlikely to succeed. We eliminated Pluronic L62 to avoid the complications a surface active agent may pose for (downstream) processes. We eliminated EDTA, nitriloacetic acid, EDTA, pyridine dicarboxylic acid, catechol, mercaptocarboxylic acid, cysteine, phenolics, and thioglycolic acid due to the organic content and their tendency to complex radionuclides. These complexants could solubilize contaminants and detract from process efficiency.

This selection process thus arrived at 0.5 M oxalic acid, 0.25 M oxalic acid, 4 M nitric acid, 0.5 M nitric acid, 0.5 M oxalic acid, and 0.5 M ascorbic acid for screening tests with simulated SRS High Level Waste supernate, sludge, and MST. Oxalic acid is the current baseline cleaning approach. Nitric acid only adds nitrate to the high level waste system. The SRS Separations

Canyons already add ascorbic acid. The panel believed citric acid would have minimal impact on the High Level Waste System. Table 1 shows the cleaning solutions tested with simulant and actual waste.

Table 1. Test Solutions and Conditions

	Simulant Test 1	Simulant Test 2	Actual Waste Test
Sludge	Simulated Tank 40 H	Simulated Tank 40H	Tank 8F
Sludge amount	5 g	5 g	1 g
MST amount	5 g	5 g	1 g
Cleaning solution amount	300 ml	300 ml	60 ml
Cleaning solutions	0.5 M oxalic acid ^{&} 0.25 M oxalic acid ^{&} 0.5 M nitric acid ^{&} 4 M nitric acid ^{&} 0.5 M citric acid ^{&} 0.5 M scorbic acid ^{&}	0.5 M oxalic acid ^{&} 4 M nitric acid ^{&} 2 M nitric acid ^{&} 1 M nitric acid ^{&} 0.5 M oxalic acid [#] 4 M nitric acid [#]	0.5 M oxalic acid^{&}4 M nitric acid^{&}2 M nitric acid^{&}
0_			

[&]amp; ambient temperature

Simulant Tests

We performed the simulant tests as follows. Personnel placed simulated SRS High Level Waste Tank 40H sludge (5 g) and MST (5 g) in a beaker. They added 300 mL of the selected cleaning agent. They stirred the beakers (see Figure 1) and periodically collected supernate samples, filtered with a 0.45 μ syringe filter, and analyzed for metals using Inductively Coupled Plasma Emission Spectroscopy (ICP-ES).

Following review of the data from these initial tests, personnel performed additional tests to examine the influence of temperature and reagent concentration. These tests examined 0.5 M oxalic acid at ambient temperature, 4 M nitric acid at ambient temperature, 2 M nitric acid at ambient temperature, 1 M nitric acid at ambient temperature, 0.5 M oxalic acid at 40 °C, and 4 M nitric acid at 40 °C. The tests used the protocol described above.

Actual Waste Tests

We performed the actual waste tests as follows. Personnel placed actual SRS High Level Waste Tank 8F sludge (1 g) and MST (1 g) in a beaker. The sludge was a dried sludge, the same sludge used in other SRTC sludge dissolution tests⁴, that was ground prior to placing in the beaker. They added 60 mL of the selected cleaning agent. They stirred the beakers and collected supernate samples periodically. They then filtered with a 0.45 μ syringe filter and analyzed for metals by ICP-ES.

^{# 40 °}C



Figure 1. Experimental Apparatus

Results

Simulant Tests

Figure 2 shows the cleaning solutions after contacting the simulated sludge and MST for one hour. The oxalic acid and citric acid solutions have a yellowish color, which results from an iron complex. The ascorbic acid solution is red, which results from a different iron complex. The nitric acid solutions are clear.

Figure 3 shows the concentration of aluminum, iron, manganese, silicon, and titanium in each of the cleaning solutions after one hour of contact. The figure also shows the calculated theoretical maximum concentration of these species if they completely dissolved. We calculated this value by multiplying the concentration of each species in the simulated sludge and MST by the mass of sludge and MST added and dividing by the volume of cleaning solution added. Table 2 shows the results of the calculation

Table 2. Theoretical Maximum Concentration of Species in Cleaning Solutions

<u>Species</u>	Simulant Tests (mg/L)	Actual Waste Tests (mg/L)
Aluminum	1430	326
Iron	5830	2300
Manganese	230	417
Silicon	200	119
Titanium	5710	8000

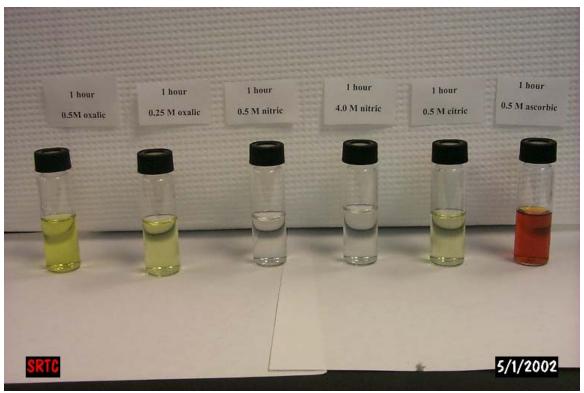


Figure 2. Cleaning Solutions after Contacting Simulated Sludge and MST for One Hour

The 0.5 M oxalic acid and 4 M nitric acid solutions dissolved almost all of the titanium. The 0.5 M oxalic acid proved most effective at dissolving iron and silicon. The 4 M nitric acid was most effective at dissolving aluminum. All of the cleaning agents dissolved about the same amount of manganese. The data shows 0.5 M oxalic acid and 4 M nitric acid to be most effective.

Based on these findings, we performed additional simulant tests with the solutions listed in Table 1. We examined lower concentrations of nitric acid in hope of discovering acceptable sludge and MST dissolution with less than 4M nitric acid. Figure 4 shows the results of that test. The figure also shows the calculated theoretical maximum concentration of these species.

The 0.5 M oxalic acid and 4 M nitric acid solutions dissolved almost all of the titanium. The 0.5 M oxalic acid proved most effective at dissolving iron and silicon. The 4 M nitric acid proved most effective at dissolving aluminum. All of the cleaning agents dissolved about the same amount of manganese. Decreasing the nitric acid concentration from 4 M to 1 M decreased the amount of iron (from 15% to 6%), aluminum (from 13% to 5%), titanium (from 88% to 33%), and silicon (from 19% to 14%) dissolved. Increasing the oxalic acid cleaning solution temperature produces a small increase for iron (from 23% to 29%) and titanium (from 98% to 100%) dissolved. Increasing the nitric acid cleaning solution temperature produces a small increase in the amount of titanium (from 88% to 97%) dissolved and a larger increase in the amount of iron (from 15% to 28%) dissolved. Increasing the cleaning solution temperature significantly increases the amount of aluminum dissolved (from 11% to 20% for oxalic acid and

from 13% to 35% for nitric acid). Again, the data shows 0.5 M oxalic acid and 4 M nitric acid are most effective.

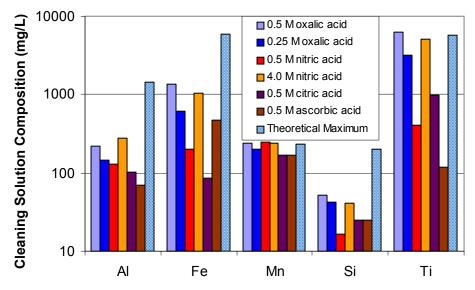


Figure 3a. Cleaning Solution Composition Following Simulant Test

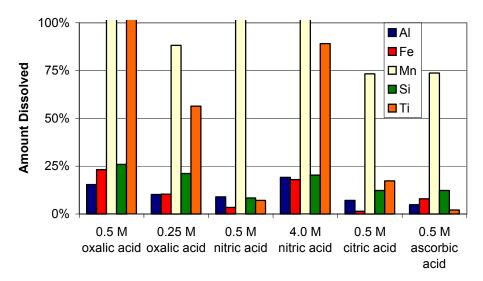


Figure 3b. Fraction of Chemical Species Dissolved in Simulant Test

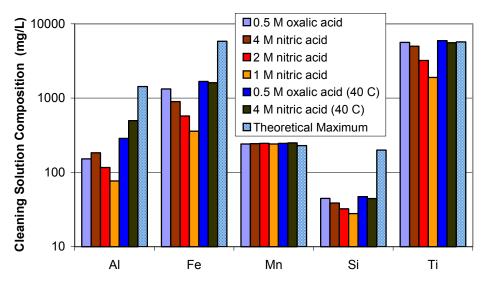


Figure 4a. Cleaning Solution Composition Following Simulant Test

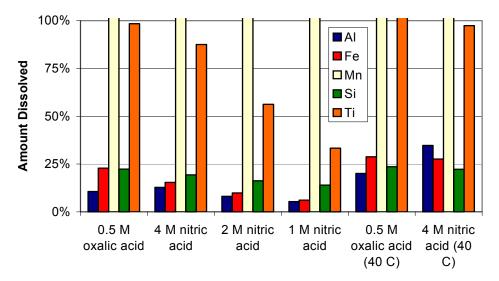


Figure 4b. Fraction of Chemical Species Dissolved in Simulant Test

We also collected samples after eight hours of contact and submitted them for analysis. Figure 5 shows the results. Increasing the contact time increased the amount of material dissolved. With oxalic acid, the increase was from 23% to 31% for iron, from 11% to 23% for aluminum, and from 98% to 100% for titanium. With 4 M nitric acid, the increase was from 15% to 28% for iron, from 13% to 36% for aluminum, and from 88% to 100% for titanium. Similar increases were observed for the other cleaning solutions. The improvement proved greatest for the components that underwent the least dissolution after one hour.

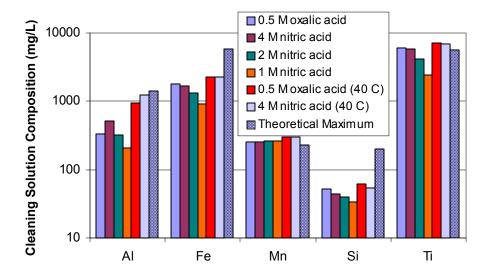


Figure 5a. Cleaning Solution Composition after Eight Hour Contact

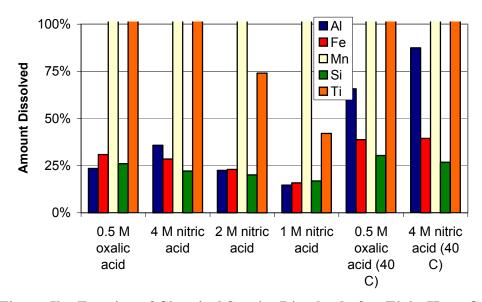


Figure 5b. Fraction of Chemical Species Dissolved after Eight Hour Contact

Actual Waste Tests

Based on the simulant test results, we decided to perform actual waste tests with 0.5 M oxalic acid, 4 M nitric acid, and 2 M nitric acid (see Table 1).

Figure 6 shows the results of the actual waste tests. The oxalic acid dissolved 100% of the titanium. The nitric acid dissolved less titanium (50-67%) than the oxalic acid. Again the oxalic acid dissolved the most iron (44%) and silicon (82%). The nitric acid dissolved slightly more aluminum (44-47%) than the oxalic acid (41%). The cleaning solutions dissolved about the same amount of manganese (63-71%).

The manganese results differ somewhat from the simulant tests. In the simulant tests, the 0.5 M oxalic acid, 4 M nitric acid, and 2 M nitric acid solutions dissolved all of the manganese. In the actual waste tests, the acids dissolved 63 - 71% of the manganese. This difference could result from differences in the manganese available to dissolve (i.e., 417 mg/L theoretical maximum in actual waste versus 230 mg/L in simulant) or to differences in the manganese compounds present in actual waste and simulant.

These tests showed 0.5 M oxalic acid as most effective at dissolving iron, silicon, and titanium. Replacing 0.5 M oxalic acid with 4 M nitric acid would increase the amount of sodium hydroxide needed for neutralization by 4X, while providing no benefit to the process. Therefore, the SPP should keep 0.5 M oxalic acid as the baseline cleaning solution.

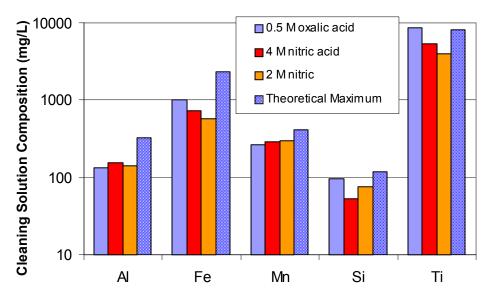


Figure 6a. Cleaning Solution Composition during Actual Waste Tests

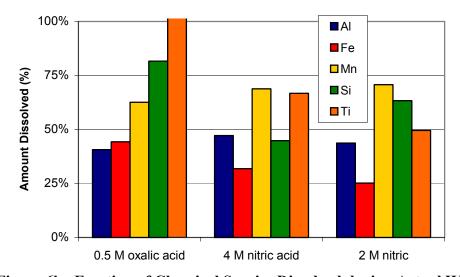


Figure 6b. Fraction of Chemical Species Dissolved during Actual Waste Tests

Conclusions

The conclusions from this work follow.

- 0.5 M oxalic acid and 4.0 M nitric acid proved most effective at dissolving iron, aluminum, and titanium. The 0.5 M oxalic acid solution dissolved 23 44 % of the iron, 11 41 % of the aluminum, and 98 100 % of the titanium calculated to be present in the sludge and MST in the tests. The 4 M nitric acid solution dissolved 15 32 % of the iron, 13 47 % of the aluminum, and 67 100% of the titanium calculated to be present in the sludge and MST in the tests. The citric acid solution dissolved 1 % of the iron, 7 % of the aluminum, and 17 % of the titanium. The ascorbic acid solution dissolved 8 % of the iron, 5 % of the aluminum, and 2 % of the titanium.
- 0.5 M oxalic acid proved more effective at dissolving iron, titanium, and silicon.
- 4 M nitric acid proved more effective at dissolving aluminum.
- Decreasing the concentration of oxalic acid or nitric acid will decrease the amount of solids dissolved. Decreasing the oxalic acid concentration to 0.25 M reduced the amount of iron (from 23 to 10 %), aluminum (from 15 to 10 %), and titanium dissolved (from 100 to 56 %). Decreasing the nitric acid concentration from 4 M to 0.5 M decreased the amount of iron (from 18 to 3 %), aluminum (from 19 to 9 %), and titanium (from 89 to 7 %) dissolved.

Recommendations

The authors recommends that the SRS Salt Processing Project keep 0.5 M oxalic acid as the baseline cleaning technology for the Salt Waste Processing Facility, and the Alpha Removal Project should use 0.5 M oxalic acid as their baseline cleaning solution.

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Appendix A - Filter Cleaning Solution Data

Simulant Test 1 Cleaning Solution Composition After One Hour Contact in mg/L

D	IIIIuIaiii	Test I Clea	ınıng Sonut	топ Сотр	osition Ait	ei Olle noi	ii Contact
		0.5 M	0.25 M	0.5 M	4.0 M	0.5 M	0.5 M
		oxalic acid	oxalic acid	nitric acid	nitric acid	citric acid	
							acid
	Αl	220	146	128	274	101	68.8
	Ва	30.6	8.92	35.4	34.6	6.87	4.90
	Ca	368	152	717	682	500	316
	Cr	0.69	0.37	0.20	0.67	0.26	0.27
	Cu	9.12	9.74	14.3	15.9	8.08	<0.05
	Fe	1350	607	201	1050	84.9	465
	Li	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	Mg	93.3	78.5	96.9	87.9	79.2	77.1
	Mn	238	203	249	236	169	170
	Мо	<0.1	<0.1	<0.1	0.10	<0.1	<0.1
	Na	3800	3600	3810	3750	3570	3610
	Ni	0.59	0.37	0.34	0.65	0.25	0.20
	Р	<0.68	1.16	<0.68	<0.68	<0.68	1.17
	Pb	14.1	<0.69	5.07	39.1	< 0.69	< 0.69
	Si	51.9	42.4	16.7	40.7	24.6	24.6
	Sr	9.35	4.47	13.6	12.9	8.26	7.03
	Ti	6160	3220	408	5090	990	119
	Zn	25.1	15.7	19.1	28.9	11.2	7.21
	Zr	0.05	<0.048	<0.048	<0.048	<0.048	<0.048
	La	<0.7	<0.7	<0.7	<0.7	<0.7	<0.7
	K	41.5	37.7	43.4	41.6	38.5	37.1

Simulant Test 2 Cleaning Solution Composition After One Hour Contact in mg/L

	0.5 M	4 M nitric	2 M nitric	1 M nitric	0.5 M oxalic	4 M nitric
	oxalic acid		acid	acid	acid (40 C)	acid (40 C)
Al	152	184	116	76.8	287	497
Ва	30.6	34.9	34.7	34.1	33.4	36.3
Ca	361	698	699	680	542	699
Cr	0.82	0.66	0.55	0.34	0.78	0.82
Cu	12.6	14.9	13.7	13.1	15.1	22.8
Fe	1330	898	575	358	1680	1610
Li	<0.2	0.20	<0.2	<0.2	<0.2	<0.2
Mg	89.1	94.5	89.4	88.2	95.7	97.4
Mn	241.5	244.1	246.1	241.6	246.5	250.3
Мо	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Na	2580	2630	2580	2570	2680	2700
Ni	0.49	0.46	0.40	0.42	0.57	0.60
Р	13	6.1	<5.0	<5.0	15.6	10.2
Pb	13.0	40.0	32.2	17.8	28.4	44.0
Si	44.7	38.7	32.4	27.9	47.1	44.5
Sr	10.0	12.8	13.0	12.8	11.5	13.2
Ti	5620	5000	3210	1900	5940	5560
Zn	24.2	24.8	21.2	18.8	29.3	36.5
Zr	<0.096	<0.096	<0.096	<0.096	<0.096	<0.096
La	<1	<1	<1	<1	<1	<1
K	47.7	44.2	42.8	39.6	42.8	42.0

Simulant Test 2 Cleaning Solution Composition After Eight Hour Contact in mg/L

	0.5 M	4 M nitric	2 M nitric	1 M nitric	0.5 M oxalic	4 M nitric
	oxalic acid	l acid	acid	acid	acid (40 C)	acid (40 C)
Al	336	511	321	210	940	1250
Ва	34.3	37.3	37.7	37.5	41.6	44.8
Ca	399	729	727	744	758	861
Cr	1.43	1.42	1.18	0.79	2.00	1.83
Cu	15.5	19.3	19.1	16.6	23.9	35.9
Fe	1800	1660	1340	923	2260	2300
Li	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Mg	96.2	103	96.7	96.8	122	125
Mn	256	256	260	260	298	303
Мо	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Na	2880	2900	2820	2910	3420	3400
Ni	0.54	0.55	0.54	0.42	0.79	0.77
Р	10.8	10.6	1.91	<1.36	13.7	12.1
Pb	18.2	44.4	42.1	30.4	44.5	56.5
Si	52.0	44.2	40.1	33.8	60.7	53.6
Sr	10.8	13.3	13.4	13.4	14.5	16.0
Ti	6030	5900	4230	2400	7240	7000
Zn	31.2	35.6	33.9	29.7	47.3	61.0
Zr	<0.096	<0.096	<0.096	<0.096	< 0.096	<0.096
La	<1.4	<1.4	<1.4	<1.4	<1.4	<1.4
K	40.7	40.3	40.3	39.0	44.2	46.1

Actual Waste Test Cleaning Solution Composition After One Hour Contact in mg/L

	0.5 M 4 M nitric		2 M nitric
	oxalic acid	acid	acid
Al	132	154	142
Ва	10	8	9
Ca	61	51	54
Cr	7	5	4
Cu	11	10	10
Fe	1018	732	579
Li	20	19	19
Mg	12	18	18
Mn	261	287	295
Мо	26	23	24
Na	3914	3801	4072
Ni	206	218	185
Р	32	30	31
Pb	15	14	14
Si	97	53	75
Sr	19	14	15
Ti	8723	5338	3964
Zn	8	5	5
Zr	17	12	10
La	12	8	9
K	428	404	424